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EDGEWOOD ARSENAL
TECHNICAL MEMORANDUM

EATM 241-2

Pyrotechnic Thermal Generation: CS Mixtures

by

Woodrow W. Reaves
Julius B. Miller

November 1966

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Task 1B522301A08101

Ground Munitions Laboratory
Weapons Development and Engineering Laboratories
US ARMY EDGEWOOD ARSENAL
Edgewood Arsenal, Maryland 21010

FOREWORD

The work described in this technical memorandum was authorized under Task 1B522301A08101, Chemical Agent Dissemination Technology. This work was started in November 1963 and completed in March 1965. The experimental data are contained in notebooks 6930 and 7101.

This memorandum is issued for temporary or limited use only, and it may be superseded.

Acknowledgements

The authors wish to acknowledge the assistance given by Analytical Chemistry Department and Field Evaluation Division for conducting the chemical analytical portion of this study.

DIGEST

The objective of this study was to devise a stable, efficient intimate mixture containing agent CS for use in various type elastomeric and small munitions. This is part of a continuing study to disseminate chemical agents from pyrotechnic mixtures.

Several of the CS intimate mixtures evaluated for this study show high vaporization efficiencies and excellent surveillance characteristics with L1 and L1U (lactose-kaolin mixtures) being two of the most promising formulations.

The surveillance studies (160°F for 90 days) with the L1-type pyrotechnic mixture indicate stability when stored in aluminum, Viton B elastomer, and butyl elastomer and unsatisfactory storage conditions when stored in natural latex containers.

Sugar-kaolin mixtures (AAK and AAKU) show equally good returns; however, only a limited number of tests were conducted with these mixtures.

TABLE OF CONTENTS

LIST OF TABLES

PYROTECHNIC THERMAL GENERATION: CS MIXTURES

I. HISTORICAL

Pyrotechnic mixtures have had a long history of useage in disseminating chemical agents. They are now the standard or most efficient means of disseminating certain standard chemical agents as colored smokes (red, yellow, green, and violet) CS, DM, CN, as well as certain experimental chemical agents.

The pyrotechnic mixture in use today consists of an oxidizing agent, potassium chlorate; a reducing agent, sugar, lactose, or sulfur; a burning rate moderator or coolant, sodium bicarbonate, magnesium carbonate, or Fuller's earth; and about 40% to 50% chemical agent.

The above mixture burns at temperatures between 300° and 800°C, and transfers heat to the chemical agent that vaporizes. These vapors leave the grenade and condense into a smoke in the relatively cool ambient air.

The first standard pyrotechnic grenade used to disseminate CS was the M7A1 CS grenade which contained an intimate pyrotechnic mixture based on thiourea and potassium chlorate as the burning mixture. This composition was unsatisfactory in that it deteriorated on storage.

This grenade was replaced by the M7A2 CS grenade, which proved to be extremely stable. The pyrotechnic mixture used in this item consisted of potassium chlorate, sugar, magnesium carbonate, cellulose nitrate, and CS in gelatine capsules.

Therefore at the time of initiation of these studies, there existed no intimate mix which combined stability on storage, high-agent content and high-vaporization efficiency. Since the completion of these studies, the M7A3 CS grenade has replaced the M7A2 CS grenade. In essence, this was only a transformation of the agent form from a gelatine encapsulation efficiency while maintaining good surveillance characteristics.

Although the above mixture is standard, it has several characteristics which precludes its use in various experimental pyrotechnic items, such as:

1. The difficulty in keeping the CS capsule or pellet uniformly dispersed in the pyrotechnic mixture results in adverse functioning qualities.

2. Loading procedure is difficult to apply to small munitions.

3. It is impossible to granulate such large pellets into 0.10 inch or small granules which is the desirable pyrotechnic form for certain exp· imental munitions.

In addition to these problems which had already become apparent, there was the unknown problem of compatibility with and efficiency of pyrotechnic mixtures in elastomeric containers that were being proposed as the basis of new and unique munitions.

II. EXPERIMENTAL

A. Materials

The list of materials used is included in the appendix.

B. Procedures

1. Selection of Mixtures to be Studied

A limited number of preliminary experiments were conducted to optimize the pyrotechnic mixtures studied. Some additional effort would probably have resulted in an increase in the efficiencies of some of the mixtures but was not conducted since the returns were excellent and the more pressing problems were degradation in surveillance and poor compatibility with elastomeric materials.

Table I indicates formulations used in this study.

2. Preparation of Intimate Agent CS-Pyrotechnic Mixtures

Prior to the blending of the pyrotechnic mixtures, several of the ingredients were screened. These materials and the sieve sizes are as follows:

a. Lactose - through a 30-mesh US standard sieve.

b. Sugar - through a 30-mesh US standard sieve.

c. Potassium chlorate - through a 60-mesh US standard sieve.

The agent CS, kaolin and magnesium carbonate were used as received.

(1) Preparation of the dry mixtures AAKU, AAMU and LIU:

The required amounts of CS, potassium chlorate, sugar or lactose, kaolin or magnesium carbonate were charged to a double cone blender and blended for 20 to 30 minutes. The blend was then screened.

Table I. Composition of Intimate CS-Pyrotechnic Mixtures

Composition designation	Ingredients (parts by weight)						
	Agent CS	Potassium chlorate	Lactose	Kaolin	Magnesium carbonate	Sugar	Nitrocellulose
AA	42	30	-	-	8	20	3.6
AAK	42	26	-	12	-	20	3.6
AAKU	42	26	-	12	-	20	-
AAMU	42	26	-	-	17	27	-
L1	42	26	20	12	-	-	3.6
L1A	40	28	20	12	-	-	3.6
L1U	42	26	20	12	-	-	-

(2) Preparation of the granulated mixtures AA, AAK, L1 and L1A:

The required amounts of CS, potassium chlorate, sugar or lactose, kaolin or magnesium carbonate were charged to a Hobart Vertical Mixer where the ingredients were mixed for 8 to 10 minutes using a flat beater. A nitrocellulose acetone solution (8 parts by weight in 92 parts by weight of acetone) was blended into the thoroughly mixed dry ingredients in the ratio of approximately 45 grams per 100 grams mixture. This procedure yielded, after drying, randomly sized granules in 8-to 20-mesh range, US standard sieve size. A minimum of materials was finer than 20 mesh. The granules' size distribution varied slightly from batch to batch.

3. Preparation and Loading of the 3-Inch Elastomeric Spheres

Four ¼ inch exhaust vents were spaced equally around the sphere, midway between the neck and the equator of the sphere. A 5/8 inch blowout patch of 0.007 inch dental dam was then cemented over these holes with rubber cement.

The neck of the sphere was spread, using a rubber stretcher tool and 100 grams of the granulated CS-pyrotechnic mixture was poured into the sphere. A 3-½ inch length of quickmatch was centered in the pyro-mix and extended into the neck of the sphere. A metal fuze adapter was then wired into place in the neck of the device.

4. Preparation and Loading of the 3-Inch Aluminum Canister

Between 65 to 80 grams of mixture (depending on the mixture used) was pressed into the aluminum canisters in three increments in a center burner configuration. Mixtures AA, AAK, L1, and L1A were pressed at 3500 pounds deadload while mixtures AAKU, AAMU, and L1U were pressed at 1500 pounds deadload. The ¼ inch center hole was primed with a slurry of starter mixture 557. The slurry was dried and the unit was capped with an aluminum dish. Quickmatch was used for ignition.

C. Results

A summary of results obtained is listed in tables II and III.

III. DISCUSSION

Upon examining table II, it is apparent that:

1. Pyrotechnic mixtures containing cane sugar (the AA series) are more effective in the thermal dissemination of CS when kaolin is present in the mixture (AAK and AAKU) then when magnesium carbonate is used (AA and AAMU).

Table II. Summary of Results

Run No. c/	Test Munition	Mix	Surveillance days	Surveillance °F	CS return %	Burning Time sec	Burning Temp °C
712	Sphere, latex, 3-in.	AA	184	Ambient	79	17	727
713	Sphere, latex, 3-in.	AA	184	Ambient	74	[illegible]	683
714	Sphere, latex, 3-in.	AA	a/		73	13	692
715	Sphere, latex, 3-in.	AA	a/	Ambient	79	13	696
718	Sphere, latex, 3-in.	AA	50	Ambient	70	13	692
719	Sphere, latex, 3-in.	AA	50	Ambient	70	15	730
724	Sphere, latex, 3-in.	AA	63	Ambient	79	16	789
725	Sphere, latex, 3-in.	AA	63	120	68	12	793
726	Sphere, latex, 3-in.	AA	63	140	51	7	721
727	Sphere, latex, 3-in.	AA	63	160	29	4	566
751	Canister, aluminum, 3 X 1-3/8 in.	AA	90	160	76	20	776
743	Canister, aluminum, 3 X 1-3/8 in.	AA	90	160	78	20	740
747	Canister, aluminum, 3 X 1-3/8 in.	AA	90	160	78	20	714
CI26	Canister, aluminum, 3 X 1-3/8 in.	AA	90	160	73	19	
CI27	Canister, aluminum, 3 X 1-3/8 in.	AA	90	160	73	21	
739	Canister, aluminum, 3 X 1-3/8 in.	AAK	90	160	100	35	682
752	Canister, aluminum, 3 X 1-3/8 in.	AAK	90	160	98	28	664
744	Canister, aluminum, 3 X 1-3/8 in.	AAK	133	Ambient	99	25	605
CI11	Canister, aluminum, 3 X 1-3/8 in.	AAK	90	Ambient	94	19	
CI17	Canister, aluminum, 3 X 1-3/8 in.	AAK	90	160	99 b/	15	
CI18	Canister, aluminum, 3 X 1-3/8 in.	AAK	90	160	63 b/	20	
CI14	Canister, aluminum, 3 X 1-3/8 in.	AAKU	90	Ambient	96	26	
CI19	Canister, aluminum, 3 X 1-3/8 in.	AAKU	90	160	93	18	

NOTE: The 3-in. canisters had a 3/8 in. vent at one end.

a/ Mix was aged in Viton for 2 months at 140°F put into latex spheres for testing.
b/ Burned with irregular emission.
c/ CI refers to tests conducted by Field Evaluation Division at Wind Tunnel Facility. All other tests conducted in Ground Munitions Laboratory test chamber.

Table II. (Cont'd)

Run No. c/	Test munition	Mix	Surveillance days	Surveillance °F	CS return %	Burning Time sec	Burning Temp °C
753	Canister, aluminum, 3 X 1-3/8 in.	AAMU	90	160	62	60	683
732	Canister, aluminum, 3 X 1-3/8 in.	AAMU	90	160	58	50	650
740	Canister, aluminum, 3 X 1-3/8 in.	AAMU	90	160	62	60	680
745	Canister, aluminum, 3 X 1-3/8 in.	AAMU	111	Ambient	66	40	630
764	Sphere, latex, 3-in.	L1	90	160	35	5	516
765	Sphere, latex, 3-in.	L1	91	Ambient	78	26	654
766	Sphere, latex, 3-in.	L1	91	Ambient	80	24	600
767	Sphere, latex, 3-in.	L1	90	150	45	5	537
768	Sphere, latex, 3-in.	L1	90	150	53	6	560
769	Sphere, latex, 3-in.	L1	90	160	55	6	585
770	Sphere, latex, 3-in.	L1	90	160	50	5	493
CI1	Sphere, latex, 3-in.	L1	90	160	44	5	
CI2	Sphere, latex, 3-in.	L1	90	160	53	5	
CI3	Sphere, latex, 3-in.	L1	90	160	48	5	
754	Canister, aluminum, 3 X 1-3/8 in.	L1	90	160	[illegible]	32	632
748	Canister, aluminum, 3 X 1-3/8 in.	L1	90	160	99	33	
746	Canister, aluminum, 3 X 1-3/8 in.	L1	111	160	95	30	605
741	Canister, aluminum, 3 X 1-3/8 in.	L1	110	Ambient	100	60	660
CI22	Canister, aluminum, 3 X 1-3/8 in.	L1	90	160	93	33	
CI23	Canister, aluminum, 3 X 1-3/8 in.	L1	90	160	97	36	
CI12	Canister, aluminum, 3 X 1-3/8 in.	L1	90	Ambient	91	31	
2	Sphere, Butyl rubber, 3-in.	L1	Control		85		
2	Sphere, Butyl Rubber, 3-in.	L1	Control		81		

NOTE: The 3-in. canisters had a 3/8 in. vent at one end.

a/ Mix was aged in Viton for 2 months at 140°F put into latex spheres for testing.
b/ Burned with irregular emission.
c/ CI refers to tests conducted by Field Evaluation Division at Wind Tunnel Facility. All other tests conducted in Ground Munitions Laboratory test chamber.

Table II. (Cont'd)

Run No. c/	Test munition	Mix	Surveillance days	Surveillance °F	CS return %	Burning Time Sec	Burning Temp °C
3	Sphere, Butyl rubber, 3-in.	L1	187	Ambient	90		
4	Sphere, Butyl rubber, 3-in.	L1	90	160	71		
5	Sphere, Butyl rubber, 3-in.	L1	90	160	80		
6	Sphere, Butyl rubber, 3-in.	L1	90	160	81		
7	Sphere, Butyl rubber, 3-in.	L1	90	160	74		
755	Canister, aluminum, 3 X 1-3/8 in.	L1A	155	Ambient	100	25	681
733	Canister, aluminum, 3 X 1-3/8 in.	L1A	90	160	87	45	663
737	Canister, aluminum, 3 X 1-3/8 in.	L1A	90	160	94	25	563
749	Canister, aluminum, 3 X 1-3/8 in.	L1A	90	160	91	42	
CI9	Canister, aluminum, 3 X 1-3/8 in.	L1A	90	Ambient	81	49	
CI15	Canister, aluminum, 3 X 1-3/8 in.	L1A	90	160	88	24	
CI16	Canister, aluminum, 3 X 1-3/8 in.	L1A	90	160	88	31	
756	Canister, aluminum, 3 X 1-3/8 in.	L1U	90	160	100	40	605
738	Canister, aluminum, 3 X 1-3/8 in.	L1U	90	160	103	65	598
742	Canister, aluminum, 3 X 1-3/8 in.	L1U	112	Ambient	100	60	600
CI13	Canister, aluminum, 3 X 1-3/8 in.	AAMU	90	Ambient	51	39	
CI24	Canister, aluminum, 3 X 1-3/8 in.	AAMU	90	160	53	40	
CI25	Canister, aluminum, 3 X 1-3/8 in.	AAMU	90	160	53	55	
715	Sphere, Viton B, 3-in.	L1	43	Ambient	70	25	615
706	Sphere, Viton B, 3-in.	L1	43	Ambient	70	29	615
704	Sphere, Viton B, 3-in.	L1	43	Ambient	70	39	587
708	Sphere, Viton B, 3-in.	L1	46	Ambient	86	29	512
709	Sphere, Viton B, 3-in.	L1	46	Ambient	86	25	500

NOTES: The 3-in. canisters had a 3/8 in. vent at one end.

a/ Mix was aged in Viton for 2 months at 140°F put into latex spheres for testing.
b/ Burned with irregular emission.
c/ CI refers to tests conducted by Field Evaluation Division at Wind Tunnel Facility. All other tests conducted in Ground Munitions Laboratory test chamber.

Table II. (Cont'd)

Run No. c/	Test munition	Mix	Surveillance days	Surveillance °F	CS return %	Burning Time sec	Temp °C
771	Sphere, Viton B, 3-in.	L1	90	160	81	12	615
772	Sphere, Viton B, 3-in.	L1	90	160	75	11	598
773	Sphere, Viton B, 3-in.	L1	92	Ambient	76	35	575
774	Sphere, Viton B, 3-in.	L1	92	Ambient	80	28	560
775	Sphere, Viton B, 3-in.	L1	92	Ambient	81	35	587
776	Sphere, Viton B, 3-in.	L1	90	160	74	13	543
777	Sphere, Viton B, 3-in.	L1	90	160	83	15	572
778	Sphere, Viton B, 3-in.	L1	90	160	81	13	531
779	Sphere, Viton B, 3-in.	L1	90	160	81	11	579
780	Sphere, Viton B, 3-in.	L1	90	160	79	10	536
CI4	Sphere, Viton B, 3-in.	L1	90	160	79	10	
CI5	Sphere, Viton B, 3-in.	L1	90	160	74	12	
CI6	Sphere, Viton B, 3-in.	L1	90	160	76	11	
CI7	Sphere, Viton B, 3-in.	L1	90	Ambient	91	39	
CI8	Sphere, Viton B, 3-in.	L1	90	Ambient	93	38	
710	Sphere, Viton B, 3-in.	L1	48	Ambient	88	34	584
711	Sphere, latex, 3-in.	L1	48	Ambient	86	25	606
716	Sphere, latex, 3-in.	L1	50	Ambient	94	21	652
717	Sphere, latex, 3-in.	L1	50	Ambient	94	24	590
761	Sphere, latex, 3-in.	L1	90	Ambient	82	25	615
762	Sphere, latex, 3-in.	L1	90	Ambient	88	26	608
763	Sphere, latex, 3-in.	L1	90	160	42	5	590
750	Canister, aluminum, 3 X 1-3/8 in.	L1U	90	160	100	50	

NOTE: The 3-in. canisters had a 3/8 in. vent at one end.

a/ Mix was aged in Viton for 2 months at 140°F put into latex spheres for testing.
b/ Burned with irregular emission.
c/ CI refers to tests conducted by Field Evaluation Division at Wind Tunnel Facility. All other tests conducted in Ground Munitions Laboratory test chamber.

Table II. (Cont'd)

Run No. c/	Test munition	Mix	Surveillance days	Surveillance °F	CS return %	Burning Time sec	Burning Temp °C
C110	Canister, Aluminum, 3 X 1-3/8 in.	LIU	90	Ambient	90	120	
C120	Canister, Aluminum, 3 X 1-3/8 in.	LIU	90	160	89	137	
C131	Canister, Aluminum, 3 X 1-3/8 in.	LIU	90	160	89	120	

NOTE: The 3-in. canisters had a 3/8 in. vent at one end.

a/ Mix was aged in Viton for 2 months at 150°F put into latex spheres for testing
b/ Burned with irregular emission.
c/ CI refers to tests conducted by Field Evaluation Division at Wind Tunnel Facility. All other tests conducted in Ground Munitions Laboratory test chamber.

Table III. Statistical Evaluation of L1-Type Pyrotechnic Mix Surveillance Data

Material	Surveillance °F	No. of Samples	Average vaporization efficiency with variance %
Viton B	160	10	78 ±3
	Control	10	80 ±8
Aluminum	160	16	95 ±5
	Control	6	94 ±8
Butyl	160	4	77 ±5
	Control	3	85 ±5

NOTE: Applying the t test to the butyl surveillance data, the null hypothesis is accepted at the 95% confidence level.

2. These sugar-kaolin mixtures show equally good agent returns as those mixtures containing lactose-kaolin (L1, L1A, L1U).

Due to limitations in time and personnel, it was decided that the work be concentrated on L1 and L1U.

A consideration of data presented in table III indicates that after storage for approximately 90 days at 160°F, the L1-type mix showed no deterioration when stored in Viton B or in aluminum. The results of storage of L1-type mix in butyl elastomer are not as clear cut because of the limited number of samples evaluated; but if the control results can be considered to be the same from butyl as Viton B, which seems likely as they are the same size and shape units, or if one utilizes the statistical t test, there is no degradation under these conditions either.

It should also be noted that L1 mix stored in the 3 inch natural rubber-latex spheres for approximately 90 days at 160°F gave returns between 35% to 55%, which are sharp decreases in vaporization efficiencies. It is believed at this time that the CS permeated the latex spheres as evidence by the appearance of CS crystals on the outer surface of these spheres. To what extent this diffusion of CS through the latex wall occurs and its effect on the agent return is not known nor is it being investigated because of availability of better elastomers.

Under Contract No. DA18-035-AMC-289(A), United States Rubber Company developed a butyl elastomeric compound (code designation 17701-CN) which will not support combustion and which has shown great promise when stored with CS. The following table shows the stability of CS (by chemical analysis) when stored for extended periods of time at 160°F in a unit made of this elastomer. The CS-pyrotechnic mix used was the L1U mixture.

Table IV

CS Determined After Aging in Oven at 160°F in 17701-CN Elastomer

	%
Start	40.7
30 days	39.5
60 days	38.3
90 days	38.8

The data acquired in this study will allow the use of an intimate pyrotechnic mixture in the relatively small submunition size listed in table II, or smaller, as well as in butyl containers. Prior to these studies, there were no completely satisfactory, i.e., smooth burning, efficient, stable mixtures for either of above devices.

IV. CONCLUSIONS

Several of the CS-intimate mixtures evaluated for this study show high-vaporization efficiencies and excellent surveillance characteristics with L1 and L1U (lactose-kaolin mixtures) being two of the most promising formulations.

The surveillance studies (160°F for 90 days) with the L1-type pyrotechnic mixture indicate stability when stored in aluminum, Viton B elastomer, and butyl elastomer and unsatisfactory storage conditions when stored in natural latex containers.

Sugar-kaolin mixtures (AAK and AAKU) show equally good returns; however, only a limited number of tests were conducted with these mixtures.

APPENDIX

Materials Used in this Study

Acetone, technical grade	Federal Specification O-A-51b, 13 Aug 64
Chemical agent, CS (orthochlorobenzalmalcnitrile)	Specification MIL-C-51029 (CmlC) 30 June 60
Chemical agent, CS	Specification MIL-C-50090A (CmlC) 21 Feb 62
Kaolin, N. F.	Fisher Scientific Company Silver Spring, Maryland
Lactose, technical	Specification MIL-L-13751 (CmlC) 4 Nov 54
Magnesium carbonate	Specification MIL-M-11361B, 25 May 62
Nitrocellulose, grade D	Specification JAN-N-244, 31 Jul 45
Potassium chlorate, technical, grade B, class 3	Specification MIL-P-150B, 8 Aug 62
Sugar, refined, cane	Specification JJJ-S 791d, 2 Nov 60
Quickmatch	Specification JAN-Q378
Rubber Dental Dam, 0.007 inch	Hygienic Dental Mfg. Co. Akron 10, Ohio
Rubber Sphere, butyl, 1/16 inch thick, No. BA63A	Pelmor Laboratory, Inc. Newtown, Pennsylvania
Rubber sphere, Viton-B, 1/16 inch thick	Pelmor Laboratory, Inc. Newtown, Pennsylvania
Rubber sphere, latex, 1/8 inch thick	H. B. Hirsch Co. & Son 91 E. Barre Street Baltimore, Maryland

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
Chemical-agent dissemination	N/A

13. ABSTRACT

The objective of this study was to devise a stable, efficient, intimate pyrotechnic mixture containing agent CS for use in various types of elastomeric and small munitions. A number of intimate CS pyrotechnic mixtures were prepared and loaded into 3-in. elastomeric spheres or 3-in. aluminum canisters. These units were placed into surveillance at either ambient temperatures or 160° F for various time intervals. Pyrotechnic mixtures containing cane sugar are more effective in the tunnel dissemination of CS when kaolin is present in the mixture than when magnesium carbonate is used. These sugar-kaolin mixtures show equally good returns as those mixtures containing lactose-kaolin. A number of CS-intimate mixtures evaluated for this study show high vaporization efficiencies and excellent surveillance characteristics. Surveillance studies with the agent CS, lactose-kaolin pyrotechnic mixture indicate stability when stored in aluminum, Viton B elastomer and butyl elastomer and unsatisfactory storage conditions when stored in natural latex containers.

14 KEYWORDS

CS mixtures	Pyrotechnic mixtures	Elastomeric munitions
Cane sugar	Magnesium carbonate	Potassium chlorate
Kaolin	Sugar-kaolin	Lactose-kaolin
Cellulose nitrate	Pyrotechnic thermal generation	

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